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Speciation of Mercury in Fish using HPLC-ICP-MS

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Introduction

The accumulation of mercury in biological samples is of interest because fish is an important compound in the human diet. So the identification and quantification of mercury species in fish plays a significant role in food safety.
When considering the toxicity of mercury the importance of the determination of the specific chemical forms from this element becomes evident. The most toxic form of mercury found in the environment is methyl mercury (CH_3Hg^+) which is formed from inorganic mercury (Hg^{2+}).
We focused on the identification of methyl mercury, ethyl- and inorganic mercury in fish samples.

To analyze the quality/toxicity of fish samples a method for mercury speciation was developed bases on HPLC-ICP-MS. The calibration method Speciated Isotope Dilution Mass Spectrometry (SIDMS) was applied.

HPLC-ICP-MS setup used for speciation

Sample pre-treatment

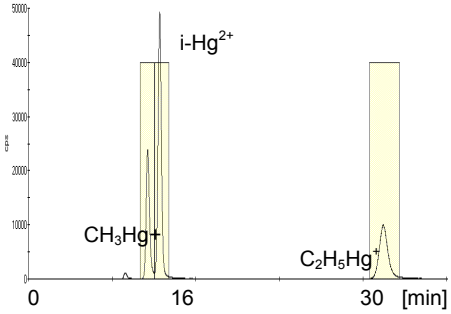
Microwave extraction I
- 200 mg sample with 25% TMAH in methanol
- In microwave at 60°C for 3 min

Microwave extraction II
- 200 mg sample with 25% TMAH in methanol
- In microwave at 60°C for 7 min

Dilution of sample extract to 50 ml with HPLC eluent

Instrumentation

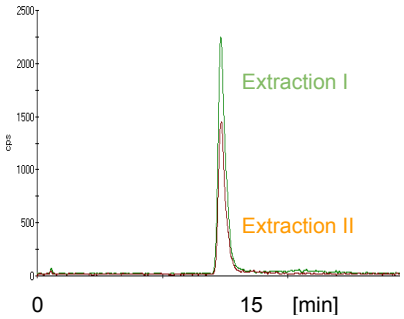
The separation of methyl mercury, ethyl- and inorganic mercury was achieved by Thermo Fisher Scientific;
Column: Synergi 4µm Hydro-RP 80A;
Injection volume: 100 µl
Eluent: 5% (v/v) methanol; 0.01% (v/v) 2-mercaptoethanol; 60 mMol ammonium acetate; ICP-MS: (Thermo Xseries II):
RF 1400 W; Cones: Platinum;
Nebulizer: Concentric.



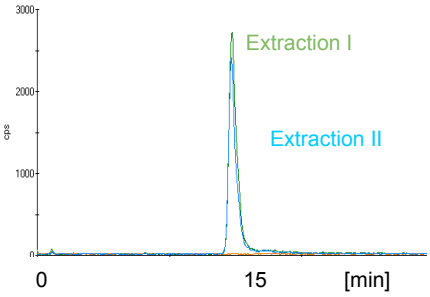
Chromatogram of standard solution i-Hg2+, CH3Hg+ and C2H5Hg+ of 100 µg/l

HPLC-ICP-MS Results

The chromatograms below show the result of the reference materials measured with HPLC-ICP-MS.



BCR 463 microwave extraction I and II



BCR 464 microwave extraction I and II

Recovery of extractions I and II are for CH_3Hg^+ in BCR-46 between 73 and 92 % and for BCR-463 between 98 and 112 % respectively.

Conclusion

HPLC-ICP-MS is a fast technique for identification of mercury species in fish samples. Interconversion between species can be corrected using SIDMS.

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